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DETERMINATION OF BORON AND LITHIUM BY RECORDING THE PRODUCTS FROM (n, α) REACTIONS

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16. Abstract

The use of nuclear reaction Li and B in solids were detd. non-destructively by irradn. with thermal neutrons in the VVR-S reactor. The charged particles produced in the reactions $Li^6(n, \alpha)H^3$ and $B^{10}(n, \alpha)Li^7$ were detected by using CsI(Tl) single crystal. For α-particle spectrometry in the B detn., an ionization chamber (W and Sn electrodes, 99% Ar + 1% H_2) was developed allowing both abs. and relative measurements. In detg. B in Li-contg. samples both scintillation and ionization chambers are used. In detg. Li in minerals, the error was 15% and the sensitivity 5×10^{-5} wt. %. In the detn. of B in SiC concn. of B $\sim (3 \pm 2) \times 10^{19}$ cm^{-3}) the error given by the α-range uncertainty was 15%.

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DETERMINATION OF BORON AND LITHIUM BY RECORDING THE PRODUCTS FROM (n, α) REACTIONS

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The neutron-activation determination of boron and lithium in various materials is difficult because they do not have isotopes which decay with a half-life suitable for analytical purposes and which have a large activation cross-section. Therefore, the reaction products from $Li^6(n, \alpha)H^3$ and $B^{10}(n, \alpha)Li^7$ reactions for thermal neutrons are used directly for the analysis of various materials. For example, the isotopic composition of lithium compounds is determined by recording the charged particles from the $Li^6(n, \alpha)$ reaction [1, 2] and the $B^{10}(n, \alpha)Li^7$ reaction is used to determine boron in silicon single crystals [3] and in steels [4]. Boron is also determined by recording the gamma radiation from the excited Li^7 nucleus [5].

However, the existing analytical methods have a number of faults: they are only suitable, in the majority of cases, for the analysis of boron or lithium in a single or a few bases. In using the method, a standard of the same material as the sample is needed and the total or partial destruction of the sample is necessary. Therefore, much attention is still being given to the development of methods for the analysis of lithium and boron.

In this study, a method is described for the determination of lithium and boron in solids without destruction of the base material, and using standards of any suitable material. Boron can also be determined by the absolute method.

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*Numbers in margin indicate pagination in foreign text.

One of the horizontal channels of the VVR-S reactor was the source of the thermal neutrons. The charged particles were detected in a CsI (tl) crystal and with an ionization chamber. The tritons from the reaction $Li^6(n,\alpha)H^3$, which have an energy of 2.73 MeV, were detected with a scintillation crystal. The alpha particles from this reaction E_α = 2.05 MeV) have a path in the sample material which is one order of magnitude less than the path for the tritons. Therefore, it is impossible to isolate the α-particles from the overwhelming triton background in the spectrum recorded by the crystal. Moreover, the conversion efficiency of the crystals is greater for tritons than for the α-particles and therefore the 2.05 MeV energy level for the α-particles corresponds to an energy of 1.5 MeV for the tritons. The α-particles do not affect the result for determining the discrimination threshold for tritons at the 1.5 MeV level.

The use of the CsI(tl) crystal for recording α-particles with energies $\leq$ 1.5 MeV is not reasonable, because of the high background which is observed in this range.

The CsI(Tl) crystal, having a diameter of 30 mm and a thickness of 20-30 mm (this thickness was chosen to avoid β- and γ-backgrounds) is placed in a light guide in the vacuum chamber (to avoid the absorption of the tritons' energy by the air) parallel to the axis of the neutron /1005
beam at a distance of 38 mm from the center of the beam. The samples were placed in the beam at an angle of 30° to its axis. The sample holder's construction allowed five samples to be measured in sequence without destroying the vacuum.

A pulsed ionization chamber was developed for the α-particle spectrometry for the $B^{10}(n, \alpha)Li^7$ reaction. Its construction was such that it operated directly in the beam from the horizontal reactor channel. Particular attention was given to the selection of the material for the electrodes and working gas in the chamber. Tungsten and tin, on which no reactions take place with the emission of charged particles were used to produce the electrodes for the chamber and the working gas was a mixture of 99% Ar + 1% N_2. The discrimination threshold was set at a 1 MeV level.

The method of modulating the frame of the thermal neutrons [6] was used directly in the measuring process to subtract the background spectrum from the mixed spectrum obtained from analyzing the sample.

In determining lithium with a scintillation detector, the measurements were made by the relative method using standards since it is difficult to account for the geometry of the sample and detector and for the topography of the neutron beam mathematically. The following formula was derived for the calculations in this case

$$N_{Li} = \frac{n_t}{n_{t_0}} \cdot \frac{(R - R_d)_0}{R - R_d} N_{Li_0},$$

where N_{Li} and N_{Li_0} are the concentration of lithium in the sample and in the standard, $(R - R_d)$ and $(R - R_d)_0$ are the difference in the paths for tritons with a maximum energy and with an energy equal to the discrimination threshold in the sample material and in the standard, n_t and n_{t_0} are the counting rates for the tritons for measuring the sample and the standard in the selected energy range referred to a unit of area.

In working with an ionization chamber, the measurements can be made by either the relative or the absolute method. In the latter case, the formula for the calculation has the form

$$N_B = \frac{4n_{\alpha}}{f \cdot \sigma \cdot \Phi (R - R_d)},$$

where f is the distribution of the B^{10} isotope, σ is the reaction cross-section, Φ is the flux of thermal neutrons, $R - R_d$ is the difference in the paths for α-particles with $E_\alpha = 1.47$ MeV and with an energy equal to the discrimination threshold.

A relationship which relates the paths of the tritons and the α-particles in the material with the path for protons [7] was used to determine the paths for the tritons and α-particles. For the difference $R - R_d$, this relationship gives a deviation from the experimental data for different materials which does not exceed 10%.

Often the problem arises of determining boron in the presence of lithium in the samples. In this case, the concentration of lithium is determined first in a scintillation chamber, in which boron does not interfere with the determination of lithium by the detector. Then the sample is placed in an ionization chamber and the total counting rate, $n_{\text{общ}}$ is determined. After the measurements are complete the concentration of boron is calculated from the formula

$$N_{\mathrm{B}} = \frac{4n_{\text{общ}}}{\sigma_{\mathrm{B}}\Phi(R-R_d)_{\mathrm{B}}} - \frac{\sigma_{\mathrm{Li}}}{\sigma_{\mathrm{B}}}\,\frac{(R-R_d)_{\mathrm{Li}}}{(R-R_d)_{\mathrm{B}}}\,N_{\mathrm{Li}}.$$

Here, $(R - R_d)_{Li}$ is the difference in the paths for the α-particles from the lithium (the energy loss for the tritons in the working volume of the ionization chamber is less than the discrimination level for the α-particles).

The results of determining boron in some silicon carbide samples are given in Table 1: /1006

Table 1: DETERMINATION OF BORON IN SILICON CARBIDE SAMPLES

Sample Number	Plate Area cm^2	Boron concentration, $cm^{-3} \times 10^{19}$ Absolute measurement	Relative measurement	Relative difference in the values, %
1	0.45	6.07	5.86	3.5
2	0.49	5.93	5.73	3.4
3	0.45	4.27	4.13	3.4
4	0.62	5.13	4.96	3.4
5	0.48	1.14	1.10	3.6

At these concentrations, the relative error is determined mainly by the inaccuracy in knowing the paths for the α-particles and is equal to ∿ 15%.

The results for determining boron and lithium in some minerals are given in Table 2.

The error in determining the lithium in the minerals is 15%, since the samples have an irregular geometric shape and the error in determining the area was 10%. The sensitivity for determining lithium and

Table 2: DETERMINATION OF BORON AND LITHIUM IN SOME MINERALS.

Sample Number	Sample Area, cm^2	Time of Measuring, sec	Lithium concentration wt. %	Boron concentration wt. %
1	3.0	120	0.0093	0.011
2	5.6	120	0.0094	0.011
3	1.75	120	0.079	0.025
4	4.75	120	0.053	0.013
5	5.1	120	0.078	0.008

boron is $\sim 5 \times 10^{-5}$ wt. %. Boron is determined in the same way as in alloyed silicon. Lithium is determined in the same way as in natural quartz and in dry residues from subterranean waters.

The described methods for determining lithium and boron allow us to study the distribution of the impurity in the volume by means of the layer-by-layer method without a loss of sensitivity. Specifically, the diffusion distribution of boron was studied in silicon carbide [8].

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